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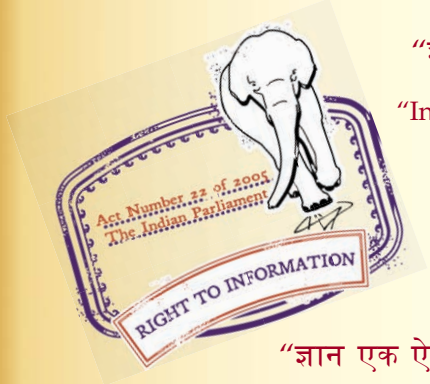

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IS : 853 - 1964
(Reaffirmed 2001)

Indian Standard

SPECIFICATION FOR BONE-MEAL, RAW

(Revised)

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BUREAU OF INDIAN STANDARDS
MANAK BHAVAN, 9 BAHADUR SHAH ZAFAR MARG
NEW DELHI 110002

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May 1965

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SPECIFICATION FOR BONE-MEAL, RAW

(Revised)

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SPECIFICATION FOR BONE-MEAL, RAW

(Revised)

0. FOREWORD

0.1 This Indian Standard (Revised) was adopted by the Indian Standards Institution on 10 December 1964, after the draft finalized by the Acids and Fertilizers Sectional Committee had been approved by the Chemical Division Council.

0.2 'Indian Standard specification for bone-meal, raw' (IS : 853-1956) was published in 1956. Suggestions were received that suitable modifications were required to protect the consumer against adulteration of bone-meal, raw, with rock phosphate, urea and sand. The Sectional Committee responsible for the preparation of this standard decided to prescribe in this standard limits of acid-insoluble matter while the requirement for nitrogen content has been made applicable for water-insoluble portion.

0.3 For the purpose of deciding whether a particular requirement of this standard is complied with, the final value, observed or calculated, expressing the result of a test or analysis, shall be rounded off in accordance with IS : 2-1960*. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

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1. SCOPE

1.1 This standard prescribes the requirements for bone-meal, raw, used as a fertilizer.

2. REQUIREMENTS

2.1 Description — The material shall be in the form of free-flowing particles, without undue fluffy, or foreign matter.

2.2 Particle Size — The material shall pass wholly through 2·36-mm IS Sieve of which not more than 30 percent shall be retained on 850-micron IS Sieve (*see* IS : 460-1962†).

*Rules for rounding off numerical values (*revised*).
†Specification for test sieves (*revised*).

2.3 Composition — The material shall comply with the requirements given in Table 1 when tested according to the methods prescribed in Appendix A. Reference to the relevant clauses of Appendix A is given in col 4 of Table 1.

TABLE 1 REQUIREMENTS FOR BONE-MEAL, RAW

Sl No.	Characteristic	Requirement	Method of Test (Ref to Cl No. of Appendix A)
(1)	(2)	(3)	(4)
i)	Moisture, percent by weight, *Max*	8·0	A-3
ii)	Acid-insoluble matter, percent by weight, *Max*	12·0	A-4
iii)	Total phosphates (as P_2O_5), percent by weight, *Min*	20·0	A-5
iv)	Available phosphates (as P_2O_5) soluble in 2 percent citric acid solution, percent by weight, *Min*	8 0	A-6
v)	Nitrogen content of water-insoluble portion, percent by weight, *Min*	3 0	A-7

3. PACKING AND MARKING

3.1 The material shall be packed in jute bags or in such other containers as agreed to between the purchaser and the supplier.

3.2 The containers shall be securely closed and marked with the name of the manufacturer; weight of the material in the container; the guaranteed percentage by weight of total phosphates and total nitrogen; recognized trade-mark, if any; and the month and year of manufacture.

3.2.1 The containers may also be marked with the Standard Mark.

3.3 The use of the Standard Mark is governed by the provisions of *Bureau of Indian Standards Act*, 1986 and the Rules and Regulations made thereunder. The details of conditions under which the licence for the use of Standard Mark may be granted to manfucaturers or producers may be obtained from the Bureau of Indian Standards.

4. SAMPLING

4.1 The method of drawing representative samples of the material and the criteria for conformity shall be as prescribed in Appendix B.

APPENDIX A

(*Clause* 2.3)

ANALYSIS OF BONE-MEAL, RAW

A-1. QUALITY OF REAGENTS

A-1.1 Unless specified otherwise, pure chemicals and distilled water (*see* IS : 1070-1960*) shall be employed in tests.

NOTE — ' Pure chemicals ' shall mean chemicals that do not contain impurities which affect the results of analysis.

A-2. PREPARED SAMPLE

A-2.1 Mix the material well by rotating the bottle several times and transfer a portion immediately into a wide-mouth bottle and stopper it immediately. Take care that no pieces of cork or sealing wax get mixed with the material. Use this *prepared sample* for tests.

A-3. DETERMINATION OF MOISTURE

A-3.1 Procedure — Weigh accurately about 5 g of the *prepared sample* in a weighed, clean, dry squat-form weighing bottle, and dry to constant weight at 105 ± 2°C. Cool in a desiccator and weigh.

A-3.2 Calculation

$$\text{Moisture, percent by weight} = 100 \frac{W_1}{W_2}$$

where

W_1 = loss in weight in g, and

W_2 = weight in g of the *prepared sample* taken for the test.

A-4. DETERMINATION OF ACID-INSOLUBLE MATTER

A-4.1 Reagent

A-4.1.1 *Dilute Nitric Acid* — 1 : 1 (*v/v*).

A-4.2 Procedure — Weigh accurately about 2 to 3 g of the *prepared sample* into a crucible and ignite gently until all organic matter is burnt away, leaving behind grey or white ash. Cool and extract the residue twice with warm dilute nitric acid. Filter through a filter paper (Whatman No. 40 or equivalent) and wash the residue on the filter paper with water. Dry the residue in an air-oven at 150 ± 2°C for one hour.

*Specification for water, distilled quality (*revised*).

Incinerate the filter paper with the residue to constant weight in a previously weighed crucible, cool in a desiccator and weigh.

A-4.3 Calculation

$$\text{Acid-insoluble matter, percent by weight} = 100\frac{W_1}{W_2}$$

where

W_1 = weight in g of the residue, and

W_2 = weight in g of the *prepared sample* taken for the test.

A-5. DETERMINATION OF TOTAL PHOSPHATE

A-5.1 Reagents

A-5.1.1 *Concentrated Sulphuric Acid* — conforming to IS : 266-1961*.

A-5.1.2 *Sodium Nitrate or Potassium Nitrate*

A-5.1.3 *Ammonium Nitrate*

A-5.1.4 *Concentrated Nitric Acid* —conforming to IS : 264-1950†.

A-5.1.5 *Ammonium Molybdate Solution* — Dissolve 200 g of crystalline ammonium molybdate in 750 ml of water, warming if necessary. Add, with shaking, concentrated ammonium hydroxide 10 to 15 ml at a time till a clear solution is obtained. Stop adding concentrated ammonium hydroxide when the solution strongly smells of ammonia (about 100 ml of concentrated ammonium hydroxide may be required). Dilute to one litre. Filter the solution through a filter paper (Whatman No. 40 or its equivalent) and store in a stoppered bottle.

A-5.1.6 *Dilute Nitric Acid* — 2 percent (*w/v*).

A-5.1.7 *Potassium Nitrate Solution* — 3 percent (*w/v*).

A-5.1.8 *Phenolphthalein Indicator* — Dissolve 0·1 g of phenolphthalein in 100 ml of rectified spirit conforming to IS : 323-1959‡.

A-5.1.9 *Standard Sodium Hydroxide Solution* — 0·1 N.

A-5.1.10 *Standard Nitric Acid* — 0·1 N.

A-5.2 Procedure

A-5.2.1 Weigh accurately about 2 g of the *prepared sample* into a 200-ml flask. Boil after adding 20 to 30 ml of concentrated sulphuric acid.

*Specification for sulphuric acid (*revised*).
†Specification for nitric acid (Since revised).
‡Specification for rectified spirit (*revised*).

Digest after adding 2 to 4 g of sodium nitrate or potassium nitrate at the beginning of digestion and a further small quantity after the solution has become nearly colourless, or adding the nitrate in small portions from time to time (before adding sodium nitrate or potassium nitrate, allow the mixture to digest at gentle heat, if necessary, until violent reaction is over). When the solution is colourless, add 150 ml of water and boil for a few minutes. Cool the solution and then transfer it quantitatively to a 250-ml volumetric flask. Make up the volume to 250 ml.

A-5.2.2 Filter the solution in the volumetric flask through a dry filter paper into a dry container. Pipette 50 ml of the filtrate into a 250-ml beaker. Add approximately 5 g of ammonium nitrate and stir. In another clean and dry beaker, place 10 ml of concentrated nitric acid. Quickly add 10 ml of ammonium molybdate solution in the second beaker with brisk stirring. There should be no turbidity or precipitation in 5 to 10 minutes. Otherwise prepare a fresh mixture. Add this molybdate mixture to the previous solution with brisk stirring. Stir for 3 minutes avoiding contact of glass rod with the walls of the beaker. Keep the solution at 40°C for 4 to 5 hours and then at room temperature for 8 to 10 hours or preferably overnight. Filter the contents by decantation through 11-cm filter paper (Whatman No. 40 or its equivalent) or a Gooch crucible for quicker filtration. Wash the beaker and the precipitate twice with dilute nitric acid (2 percent) and then with potassium nitrate solution till free from acid. Test for freedom from acidity with blue litmus paper when Gooch crucible is used. When filtered over the funnel, the filtrate from two filterings of the paper should give a permanent pink colour with phenolphthalein and one drop of standard sodium hydroxide solution. Place the filter paper and the precipitate in the original beaker and dissolve the yellow precipitate completely in a known volume of standard sodium hydroxide solution which should be in slight excess. Add a drop of phenolphthalein indicator and titrate the excess of alkali with standard nitric acid.

A-5.3 Calculation

$$\text{Total phosphate (as } P_2O_5 \text{)} = \frac{0{\cdot}3088\ V_3\ (V_1N_1 - V_2N_2)}{V_4W}$$

where

V_3 = total volume in ml of the test solution prepared in **A-5.2.1**,

V_1 = volume in ml of standard sodium hydroxide solution added to precipitate,

N_1 = normality of standard sodium hydroxide solution,

V_2 = volume in ml of standard nitric acid used in the titration,

N_2 = normality of standard nitric acid,

V_4 = volume in ml of the aliquot taken for the test in **A-5.2.2**, and

W = weight in g of *prepared sample* taken for the test.

A-6. DETERMINATION OF AVAILABLE PHOSPHATES SOLUBLE IN 2 PERCENT CITRIC ACID SOLUTION

A-6.1 Reagents

A-6.1.1 *Citric Acid Solution* — Dissolve 20 g of citric acid in water and make up the volume to 1 litre.

A-6.1.2 *Concentrated Nitric Acid* — same as in **A-5.1.4**.

A-6.1.3 *Ammonium Molybdate Solution* — same as in **A-5.1.5**.

A-6.1.4 *Dilute Nitric Acid* — 2 percent (*w/v*).

A-6.1.5 *Potassium Nitrate Solution* — 3 percent (*w/v*).

A-6.1.6 *Phenolphthalein Indicator* — same as in **A-5.1.8**.

A-6.1.7 *Standard Sodium Hydroxide Solution* — 0·1 N.

A-6.1.8 *Standard Nitric Acid* — 0·1 N.

A-6.2 Procedure — Weigh accurately about 5 g of the *prepared sample* into a 500-ml dry Wagner flask. Make up the volume to 500-ml mark with citric acid solution. Close the flask with a suitable stopper, place it at once in rotary shaking apparatus and shake the flask at the rate of 30 to 40 rev/min for 30 minutes. At the end of this period, remove the flask, filter the solution through a dry filter paper (Whatman No. 42 or its equivalent) into a clean dry container. Reject the first few millilitres of the filtrate, if turbid. Determine the phosphate (as P_2O_5) in a suitable aliquot as under **A-5.2.2** and **A-5.3**.

A-7. DETERMINATION OF NITROGEN CONTENT OF WATER-INSOLUBLE PORTION

A-7.1 Apparatus — The apparatus as assembled is shown in Fig. 1. It consists of a flask *A* of 1 000 ml capacity fitted with a rubber stopper through which passes one end of the connecting bulb tube *B* and the end of the dropping funnel *F*. The other end of the bulb tube *B* is connected to the condenser *C* by a rubber stopper, and the lower end of the condenser *C* is attached by means of a rubber tubing to dip tube *D* into a beaker *E* of 250 ml capacity.

A-7.2 Reagents

A-7.2.1 *Potassium Sulphate* — powder.

A-7.2.2 *Concentrated Sulphuric Acid* — conforming to IS : 266-1961*.

A-7.2.3 *Copper Sulphate*

A-7.2.4 *Boric Acid Solution* — 5 percent (*w/v*).

*Specification for sulphuric acid (*revised*).

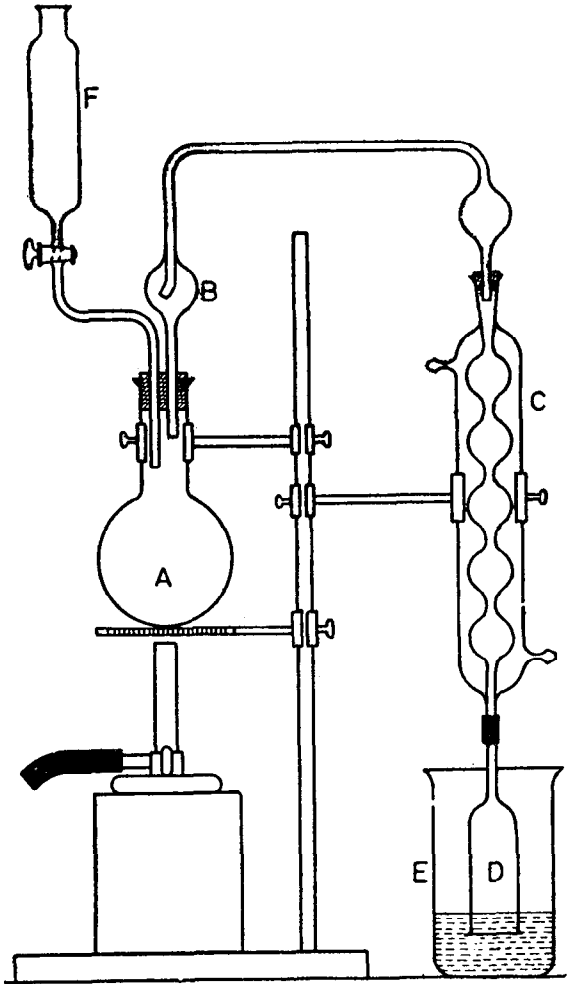

FIG. 1 ASSEMBLY OF APPARATUS FOR THE DETERMINATION OF NITROGEN

A-7.2.5 *Sodium Hydroxide Solution* — approximately 30 percent (*w/v*).

A-7.2.6 *Mixed Indicator* — Dissolve 1·2 g of methyl red and 0·825 g of methylene blue in 1 000 ml of alcohol. Store it in a dark-coloured bottle.

A-7.2.7 *Standard Sulphuric Acid* — 0·1 N.

A-7.3 Procedure — Weigh accurately about 1·5 g of the *prepared sample* into a clean beaker. Stir well with water for a few minutes and filter through a filter paper (Whatman No. 2 or equivalent). Transfer the residue completely to the filter paper using a fine jet of water. Carefully put the filter paper containing the residue into a 500-ml Kjeldahl flask. Add 10 g of potassium sulphate, 0·5 g of copper sulphate and 20 to 25 ml of concentrated sulphuric acid. Gently heat the flask keeping it in an inclined position until frothing has ceased. Increase heat until the acid boils briskly, digest till the mixture becomes clear and continue boiling for another one hour. Cool the flask and dilute the contents of the flask to 300 ml. Pipette 30 ml of 5 percent boric acid solution into beaker *E* and assemble the aparatus as shown in Fig. 1 with the stop-cock of the dropping funnel *F* closed. Make sure that all connections are air-tight and water is flowing through the condenser. Pour carefully through the dropping funnel *F* about 150 ml of sodium hydroxide solution. Ensure that there is sufficient excess of alkali. Close the stop-cock and gently shake the flask. Gradually increase the heat to boiling. Distil till all the ammonia has passed over into the beaker *E* (first 150 ml of distillate generally contains all the ammonia). Open the stop-cock of the dropping funnel to prevent a suck back, shut off the burner, and when the flask becomes cool, detach it from the condenser. Lower the beaker *E* until the end of the dip tube *D* is out of the acid and rinse the condenser thoroughly with water into the beaker *E*. Add two or three drops of mixed indicator and titrate with standard sulphuric acid. This mixed indicator gives violet colouration in acid medium and green in alkaline. Dim blue colour gives the end point.

A-7.3.1 Carry out a blank test using all the reagents in the same quantities but without the *prepared sample*.

A-7.4 Calculation

$$\text{Nitrogen content of water-insoluble portion, percent by weight} = \frac{1{\cdot}4008\,(B-A)\,N}{W}$$

where

B = volume in ml of standard sulphuric acid solution used in the blank test,

A = volume in ml of standard sulphuric acid used in the test with the *prepared sample*,

N = normality of standard sulphuric acid, and

W = weight in g of the *prepared sample* taken for the test.

APPENDIX B

(*Clause* 4.1)

SAMPLING OF BONE-MEAL, RAW

B-1. GENERAL REQUIREMENTS OF SAMPLING

B-1.0 In drawing, preparing, storing and handling samples, the following precautions and directions shall be observed.

B-1.1 Precaution shall be taken to protect the samples, the material being sampled, the sampling instrument and the containers for samples from adventitious contamination.

B-1.2 Samples shall be placed in clean and air-tight glass or other suitable containers.

B-1.3 Each sample container shall be sealed air-tight after filling and marked with full details of sampling and the date of sampling.

B-2. SCALE OF SAMPLING

B-2.1 Lot — In any consignment, all the containers of the same size and drawn from the same batch of manufacture shall constitute a lot. If a consignment is known to consist of different batches of manufacture and of different sizes of containers, the containers belonging to the same batch and size shall be grouped together and each such group shall constitute a separate lot.

B-2.2 For ascertaining the conformity of the material in the lot to the requirements of this specification, tests shall be carried out for each lot separately. The number (n) of containers to be selected for this purpose shall depend on the size of the lot (N) and shall be in accordance with Table 2.

TABLE 2 NUMBER OF CONTAINERS TO BE SELECTED FOR SAMPLING

LOT SIZE	NO. OF CONTAINERS TO BE SELECTED
N	n
(1)	(2)
4 to 15	3
16 ,, 40	4
41 ,, 65	5
66 ,, 110	7
111 and above	10

NOTE — When the size of the lot is 3 or less, the number of containers to be selected for sampling and the method of judging the conformity of the lot to the specification requirements shall be as agreed to between the purchaser and the supplier.

B-2.3 The containers shall be selected at random and to ensure randomness of selection, a random number table as agreed to between the purchaser and the supplier shall be used. In case such a table is not available, the following procedure is recommended for use:

Starting from any container, count them as 1, 2, 3,..., up to r and so on in one order. Every rth container thus counted shall be withdrawn to give sample for tests, where r is the integral part of N/n.

B-3. PREPARATION OF SAMPLES

B-3.1 From each container selected, draw with an appropriate sampling instrument a representative portion of the material not less than 250 g.

B-3.2 Out of these portions, an equal quantity of the material shall be taken and mixed together to form a composite sample of about 300 g. The composite sample so formed shall be divided into 3 equal parts, one for the purchaser, another for the supplier and the third to be used as a referee sample.

B-3.3 The remaining portion of the material from each container shall be divided into 3 equal parts, each forming an individual sample. One set of individual samples representing the n containers selected shall be marked for the purchaser, another for the supplier and the third for the referee.

B-3.4 All the individual and composite samples shall be transferred to separate moisture-proof containers and labelled with full identification particulars.

B-3.5 The referee sample consisting of a composite sample and a set of n individual samples shall bear the seals of both the purchaser and the supplier. They shall be kept at a place agreed to between the two parties, to be used in case of dispute.

B-4. NUMBER OF TESTS

B-4.1 Tests for the determination of total phosphates and nitrogen content of water-insoluble portion shall be done on each of the individual samples.

B-4.2 Tests for particle size (*see* **2.2**) and all other characteristics listed in Table 1 shall be conducted on the composite sample.

B-5. CRITERIA FOR CONFORMITY

B-5.1 For Individual Samples — For each of the characteristics tested on the individual samples, the mean ($\bar{X}$) and range (R) of test results shall be computed separately (range being defined as the difference beween the maximum and the minimum of the test results).

B-5.1.1 The lot shall be declared as conforming to the requirements for the characteristics tested on the individual samples if the conditions given below are satisfied:

a) For total phosphates $\bar{X} - 0{\cdot}6\,R \geqslant 20{\cdot}0$

b) For nitrogen content of water-insoluble portion $\bar{X} - 0{\cdot}6\,R \geqslant 3{\cdot}0$

B-5.2 For Composite Samples — For declaring the conformity of the lot to the requirements of the characteristics tested on the composite sample, the test result for each characteristic shall satisfy the relevant requirement specified.

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